THE ADSORPTION OF ARGON AND NITROGEN ON STAINLESS STEEL

AT 195°K AND 303°K IN THE PRESSURE RANGE

$$10^{-8} \text{ to } 10^{-7} \text{ TORR}$$

by

James P. Wightman

Chemistry Department
Virginia Polytechnic Institute
Blacksburg, Virginia, 24061

and

John P. Mugler, Jr.

NASA Langley Research Center
Langley Station, Hampton, Virginia, 23365

PRESENTED AT THE

45th Annual Meeting of the Virginia Academy of Science
before the Space Science and Technology Section

Norfolk, Virginia
May 5, 1967

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Langley Research Center

THE ADSORPTION OF ARGON AND NITROGEN ON STAINLESS STEEL AT

195° K AND 303° K IN THE PRESSURE RANGE 10^{-8} TO 10^{-7} TORR

by James P. Wightman and John P. Mugler, Jr.

I. INTRODUCTION

The general area of adsorption of gases on solids at low pressures
($< 10^{-6}$ torr) either has been shown or suspected to play an important role in
diverse aerospace phenomena. The cold welding of solids and the measurement
of outgassing rates of solids are cited as relevent examples. However,
essentially no definitive experimental data exists for the adsorption of gases
at low pressures on alloys. The present work is a continuation of work
initiated under the 1965 NASA-ASEE program at the NASA-Langley Research Center.
Specifically, the adsorption of argon and nitrogen on stainless steel was

investigated at 195° K and 303° K in the pressure range 10^{-8} to 10^{-7} torr.

II. EXPERIMENTAL

1. _Apparatus._- Some of the components of the apparatus used during the
1965 NASA-ASEE program were the same as used in the current program. However,
there were enough significant differences in the apparatus and procedure such
that direct referencing of reference 1 was not always possible. A schematic
diagram of the apparatus constructed for the current program is shown in
figure 1. The symbols refer to components described in table 1. The actual
apparatus is shown in the photograph in figure 2. The test system consisted
of an adsorption vessel (AV), a storage vessel (SV), and a gas inlet. The gas
inlet consisted of a variable inlet valve (V_3), a liquid nitrogen cooled trap
(Tr), a gas source (Ta), a valve (V_8), and a mechanical pump (P). The
adsorption vessel and the storage vessel were connected to a chamber (T) by
valves (V_4) and (V_5), respectively. The Orb-ion pump (OIP) (ref. 2) was

connected directly to the chamber (T). The Orb-ion pump was not functional

at pressures $> 10^{-3}$ torr as monitored by a thermocouple gage (TC). Pressures
in the system of $\leq 10^{-3}$ torr were achieved within 5 minutes by use of a liquid
nitrogen cooled sorption pump (SP_1) connected by the system through valve (V_6).

Sorption pump (SP_2) was used as a reserve in case the load in the system

exceeded the capacity of (SP_1). However, the reserve pump was not used.

The wall of the cylindrical stainless steel adsorption vessel served as the adsorbent. The volume of the adsorption vessel was 3.46 l. and the calculated geometric area was 1150 cm^2. The volumes of the doser and storage vessel were 3.51 cm^3 and 3.96 l., respectively. Adsorption measurements were made at 195° K and at 303° K. The lower temperature was achieved by surrounding the adsorption vessel completely with crushed dry ice. All temperature measurements except dry ice temperatures were made with chromel-alumel thermocouples and were recorded on a Bristol recorder. Thermocouples were located at the following points:

(1) Adsorption vessel (AV)
(2) Doser (D)
(3) Storage vessel (SV)
(4) Tubulated gage (TG$_1$)
(5) Valve (V$_4$)
(6) Chamber (T)
(7) Valve (V$_5$)
(8) Orb-ion pump (top)(OIP)
(9) Orb-ion pump (bottom)(OIP)
(10) Variable inlet valve (V$_3$)

Pressures in the storage and adsorption vessels were measured with a tubulated Veeco RG 75K ionization gage (TG$_1$) and a nude Varian UHV-14 ionization gage, (NG$_1$), respectively. Gages (TG$_2$) and (NG$_2$) were also a tubulated Veeco and nude Varian, respectively, and were placed in the system to be used in case of failure of either (TG$_1$) or (NG$_1$). The emission current was normally set at 1 ma for the Veeco gage and at 4 ma for the Varian gage. The manufacturer's recommended settings for the Veeco and Varian gages are 10 ma and 4 ma, respectively. Both gages were cleaned by electron bombardment. Typical bombing conditions were as follows: Varian - grid current, 84 ma; Veeco - filament voltage, 6 V; grid voltage, 800 V; grid current, 100 ma. The gages were bombed for 15 minutes followed by a cool-down period of at least 2 hours. No attempt was made to determine at what point in the test series gage cleanup should be initiated. The gages were cleaned at least once and sometimes twice daily. The entire test system was baked at 230° C (450° F) for 18 hours if pressures < 10^{-9} torr in the adsorption vessel were not reached readily.

2. **Gases.**- Research grade argon (99.9995 percent min.) was obtained from the Matheson Company and used without further pretreatment. Seaford grade (oil free) nitrogen was obtained from the Southern Oxygen Company and the following percentage composition noted: O$_2$ - 0.002 percent; H$_2$ - 0.05 percent; Ar - 0.06 percent; H$_2$O - 0.002 percent; N$_2$ - balance. Nitrogen was passed through a liquid nitrogen trap (Tr) prior to use.

3. **Procedure.**- In a typical experimental run for the adsorption measurements, the initial configuration would be valves (V$_2$), (V$_4$), and (V$_5$) opened, and valves (V$_1$) and (V$_3$) closed. Valve (V$_1$) was closed at 20 inch-pounds.

The pressures in the adsorption and storage vessels in the initial configuration were typically $< 5 \times 10^{-10}$ and $< 1 \times 10^{-9}$ torr, respectively. The gas inlet system was first flushed repeatedly with the test gas to insure minimum contamination. After closing valve (V_5) at an undetermined torque, gas was bled into the storage vessel through valve (V_3) until a convenient pressure was reached, typically 10^{-5} torr. The pressure in the storage vessel was monitored by gage (TG_1) and was recorded as a function of time on a calibrated Sanborn recorder (Model 150-1000). Parenthetically, the pressure-time data obtained was used to calculate the pumping speed of argon and nitrogen by gage (TG_1). Valve (V_3) was then closed and a cycle designated (1-4) was initiated whereby valve (V_4) was closed at 10 foot-pounds: step 1; valve (V_2) was closed at 30 inch-pounds: step 2; valve (V_2) was opened: step 3; and, valve (V_1) was closed then at 20 inch-pounds after 10 seconds: step 4; in that order.* Thus, the test gas in doser (D) of known volume at a pressure equal to that in the storage vessel (P_{SV}) expanded into the adsorption vessel of known volume to a final pressure (P_{AV}). The pressure in the adsorption vessel was monitored by gage (NG_1) and was recorded as a function of time on a calibrated Sanborn recorder (Model 150-1500). The measured values of P_{AV} and P_{SV} were used to calculate an expansion ratio defined as P_{SV}/P_{AV}. The extent of adsorption was inferred from the expansion ratios.

The adsorption vessel was readied for the next gas dose by opening valve (V_4) to reduce the adsorption vessel to a low base pressure, typically 3×10^{-10} torr. The pressure in the storage vessel was increased by bleeding additional gas through valve (V_3). Note that the storage vessel was not pumped down between measurements. Valve (V_5) remained closed for periods up to 1 hour.

The following supplementary measurements were made in addition to the adsorption measurements:

1. The outgassing of the adsorption vessel.
2. The contribution of the (1-4) cycle to the measured pressure in the adsorption vessel.
3. The pumping speeds of argon and nitrogen by gage (TG_1).

The initial configuration for the determination of the outgassing of the adsorption vessel was valve (V_1) closed at 20 inch-pounds and valve (V_4) opened. Valve (V_4) was then closed at 10 foot-pounds and the pressure as monitored by gage (NG_1) was recorded as a function of time. The pressure-time data was used to determine the contribution of the outgassing of the adsorption vessel as described in section III-4.

---

*The fourth step was necessary since valve (V_2) leaked even when closed allowing gas to pass between the storage and adsorption vessels. This step was not necessary in the previous work (ref. 1).

The initial configuration for the determination of the contribution of the (1-4) cycle to the measured pressure in the adsorption vessel would be valves (V_2), (V_4), and (V_5) opened and valves (V_1) and (V_3) closed. The same torques were used here as in the adsorption measurements above. The (1-4) cycle was then initiated whereby valve (V_4) was closed-step 1, valve (V_2) was closed-step 2, valve (V_1) was opened-step 3 and valve (V_1) was closed after 10 seconds - step 4, in that order. The pressure in the adsorption vessel was subsequently monitored by gage (NG_1) and was recorded as a function of time on a calibrated Sanborn recorder (Model 150-1500). The treatment of the (1-4) cycle data is discussed in section III-5.

The pumping speeds of argon and nitrogen by gage (TG_1) were determined simultaneously with the expansion ratio measurements as described above.

III. RESULTS

The results of all experiments are presented in this section. Some of the experiments were necessarily supplementary to the adsorption measurements. For the results of the adsorption measurements the reader may refer directly to sections III-7 and III-8.

1. _Temperature measurements._- The temperature of the adsorption vessel was maintained at $303 \pm 2^\circ$ K $(32 \pm 2^\circ$ C$)(90 \pm 4^\circ$ F) except when surrounded with dry ice. The temperature of the adsorption vessel at the lower temperature was then assumed to be sublimation temperature of dry ice, which is 195° K $(-78^\circ$ C$)(-108^\circ$ F). The temperature of the storage vessel was maintained at $301 \pm 1^\circ$ K $(28 \pm 1^\circ$ C$)(82 \pm 2^\circ$ F). The temperature of the chamber (T) depended upon the emission current at which the ion pump was operating. The temperature of the chamber was $322 \pm 3^\circ$ K $(49 \pm 3^\circ$ C$)(121 \pm 7^\circ$ F) and $313 \pm 3^\circ$ K $(40 \pm 2^\circ$ C$)$ $(105 \pm 5^\circ$ F) at emission current settings of 50 and 30 ma, respectively.

2. _Pressure capabilities of system._- The pressure in the system was significantly effected by the emission current at which the ion pump was run. The effect of increasing the emission current from 30 to 50 ma on the pressure in the adsorption vessel is seen in figure 3. The increase in pressure observed as the emission current was increased could be attributed to a higher outgassing rate as the temperature of the chamber (T) increased (see preceding section).

3. _Outgassing rate of the storage vessel._- The outgassing rate of the storage vessel was measured at an emission current of 1 ma on gage (TG_1) since pressure measurements in the storage vessel during the adsorption runs were also made at 1 ma. The nitrogen equivalent based value of the outgassing rate of the storage vessel at 301° K was 7.5×10^{-14} torr-l sec^{-1} cm^{-2} for a base pressure of 2×10^{-9} torr.

During the adsorption measurements, base pressures in the storage vessel prior to closing valve (V_5) ranged from 4×10^{-10} to 2×10^{-9} torr. The highest outgassing would be observed at base pressures of 2×10^{-9} torr. The storage vessel was closed for periods up to 100 min. The pressure rise due to outgassing of the storage vessel after this period could be 2×10^{-7} torr. This is two (2) decades lower than the lowest pressures at which gases were introduced into the storage vessel for the adsorption measurements. Therefore, it is concluded that outgassing of the storage vessel would not affect pressure measurements in the storage vessel during the adsorption runs.

4. <u>Outgassing of the adsorption vessel</u>.- Closing valve (V_4) produced a pressure change in the adsorption vessel at $303°$ K. A typical pressure-time trace obtained on closing valve (V_4) is shown in figure 4. The dashed line in figure 4 is not meant to indicate an actual pressure change but the time interval over which the pressure scale on the controller was changed to anticipate the pressure rise on expansion. The decrease in pressure suggests appreciable gage pumping. For purposes of comparison, pressures were noted 120 seconds after closing valve (V_4).* The value of the interpolated pressures at 120 seconds were proportional to the base pressure in the adsorption vessel prior to closing valve (V_4) as shown in figure 5. Hence after 2 min. pressures on the order of 10^{-9} torr were realized simply by closing valve (V_4).

Closing valve (V_4) also produced a pressure change in the adsorption vessel at $195°$ K. A similarly shaped pressure-time trace was obtained at the lower temperature. Values of the interpolated pressures 120 seconds after closing valve (V_4) were also proportional to the base pressure in the adsorption vessel prior to closing valve (V_4) as shown in figure 6. However, values of the interpolated pressures were significantly lower by almost a decade at a given base pressure at the lower temperature.

An entirely different trace was reported (ref. 1) on closing valve (V_4). There an increase in pressure with time was noted rather than a decrease. The author can make no further comment on this point.

5. <u>Pressure contribution of (1-4) cycle</u>.- The mechanical manipulation of valves (V_1, V_2, V_4) during the (1-4) cycle with the storage vessel evacuated produced a pressure change in the adsorption vessel at $303°$ K. A typical

---

*The choice of 120 seconds is somewhat arbitrary but is used consistently throughout this section and in sections III-5, III-7, and III-8. As a rule of thumb, the formation of a monolayer of adsorbed gas takes about 1 second at 10^{-6} torr. Thus at 10^{-8} torr as in the present work, times of the order of 100 seconds for adsorption should be observed. The choice of 120 seconds is felt to be more realistic than the extrapolation procedure outlined in reference 1. However, it should be realized that gage pumping may become an important factor over a 2 min. time interval.

pressure-time trace for the (1-4) cycle is shown in figure 7. The times at which steps 1 and 3 were initiated are indicated in figure 7. This trace is similar in shape to the trace obtained on completion of the (1-3) cycle described in reference 1. Again, pressures in the adsorption vessel were noted 120 seconds after opening valve (V_1) - the third step in the (1-4) cycle. Values of the interpolated pressures were directly proportional to the base pressures in the adsorption vessel prior to closing valve (V_4) as shown in figure 8. A comparison of figures 5 and 8 has been made graphically and is indicated by the dotted line in figure 8. Thus step 1: the closing of valve (V_4) contributes 90 percent of the total observed pressure change due to the (1-4) cycle.

The manipulation of valves in the (1-4) cycle also produced a pressure change in the adsorption vessel at 195° K. A similarly shaped pressure-time trace was obtained at the lower temperature. Interpolated values of the pressures in the adsorption vessel were obtained as described in the above paragraph and are shown in figure 9. Again values of the interpolated pressures are significantly lower by almost a factor of 3 at a given base pressure at the lower temperature. The line drawn in figure 9 is an average line of perhaps ten test series. Pressure corrections were interpolated from lines representing a single test series.

6. **Gage pumping speeds of argon and nitrogen.** - The necessary data to calculate pumping speeds was obtained routinely during the adsorption measurements hence pumping speeds were calculated for argon and nitrogen. A typical pressure-time trace used in the calculation of the pumping speed of argon is shown in figure 10. The calculated pumping speed for argon on initial exposure at 1 and 10 ma in gage (TG_1) was 1.2×10^{-3} and 8.6×10^{-3} l sec^{-1}, respectively. A typical pressure-time trace used in the calculation of the pumping speed of nitrogen is shown in figure 10. The calculated pumping speed for nitrogen on initial exposure at 1 ma in gage (TG_1) was 1.9×10^{-2} l sec^{-1}. The determined pumping speed of nitrogen and presumably other gases appears to be dependent on several factors including the length of exposure of the gage to the test gas and the pressure of the test gas as shown in figure 11. The pumping speed of nitrogen is seen to decrease with either increasing time of exposure of gage (TG_1) to nitrogen or increasing nitrogen pressure. It is not possible to separate the two effects of time and pressure from the available data. The same effect had been noted previously (ref. 1). The values of pumping speeds are thus specified for initial exposure of a gas to gage (TG_1).

7. **Adsorption measurements (expansion ratios) for argon.** - The extent of adsorption was inferred from the calculated expansion ratios. The expansion ratio was defined as the pressure in the storage vessel before expansion to the pressure in the adsorption vessel after expansion or (P_{SV}/P_{AV}). Adsorption of a test gas would result in a larger expansion ratio relative to a nonadsorbed reference gas since (P_{AV}) would be reduced on adsorption. Argon was chosen as

a reference gas. It was assumed that no physisorption of argon occurred at room temperature on stainless steel on the basis of the results reported by the author (ref. 1).

The expansion ratios (R) using argon calculated from the measured pressures in the adsorption vessel and doser are shown in table II. The temperature of the adsorption vessel (T_{AV}) at the time the expansion ratio was determined is indicated in column 3. The base pressure (P_B) in the adsorption vessel prior to closing valve (V_4) - the first step in the (1-4) cycle - is indicated in column 4. Pressures in the adsorption vessel (P_{AV}) indicated in column 6 are the recorded pressures 120 seconds after opening valve (V_1) - the third step in the (1-4) cycle. Typical pressure-time traces obtained on expansion of argon at 195° K and 303° K are shown in figures 12a and 12b. Various parts of figures 12 and 13 are labeled to aid in seeing how the results were obtained from experimental data. As noted in section III-5 the mechanical manipulation of valves (V_1), (V_2), and (V_4), with the storage vessel evacuated, introduced a significant quantity of gas into the adsorption vessel. The pressure in the adsorption vessel due to the (1-4) cycle labeled P_{14} thus had to be subtracted from values of P_{AV} to obtain corrected pressures due to only expansion of the test gas. The value of the contribution of the (1-4) cycle (P_{14}) was determined either by interpolation from figure 8 or figure 9 or by direct measurement. Directly measured values of P_{14} are marked with an asterisk (*) in column 5. Values without an asterisk (*) were interpolated from figure 8 or figure 9. Values of P_{14} were then subtracted from values of P_{AV} and pressures in the adsorption vessel corrected for the (1-4) cycle appear as P_{AV}^{corr} in column 7. The pressure of the gas in the doser (P_D) is equal to the pressure in the storage vessel (P_{SV}) and that pressure is noted in column 8. The expansion ratios (R) listed in column 9 are the ratios of P_{SV} to P_{AV}^{corr}. The calculated expansion ratio for argon at 303° K was 1400 with an average deviation of only 49 parts per 1000. The calculated expansion ratio for argon at 195° was 1350 with an average deviation of 111 parts per 1000. It is concluded from a comparison of the two expansion ratios that no significant adsorption of argon on stainless steel occurs on decreasing the temperature from 303° K to 195° K in the pressure range 10^{-8} to 10^{-7} torr.

It should be noted that the calculated value of the expansion ratio for argon at 303° K does not differ significantly from the value of 1200 reported previously (ref. 1). The agreement is gratifying especially since changes were made in the apparatus, the procedure, and the reduction of data.

The marked increase in base pressure between runs 3 and 4 of 8-8-66 is indication of the relatively slow pumping of argon by the ion pump as contrasted to nitrogen. The storage vessel was pumped out between selected runs to minimize leakage through valve (V_1) and (V_2) to the adsorption vessel and to

permit determination of the pressure contribution of the (1-4) cycle between adsorption measurements. However, this procedure was not followed for argon due to the slow pumping speed of the ion pump.

8. <u>Adsorption measurements (expansion ratios) for nitrogen</u>.- The expansion ratios (R) using nitrogen calculated from the measured pressures in the adsorption vessel and doser are shown in tables III and IV. Typical pressure-time traces obtained on expansion of nitrogen at 195° K and 303° K are shown in figures 13a and 13b. The data for nitrogen was treated in a similar manner as the data for argon so all columns in tables III and IV have counterparts in table II. The calculated expansion ratios for nitrogen at 303° K was 22,000 with an average deviation of 200 parts per 1000. The calculated expansion ratio for nitrogen at 195° K was 24,500 with an average deviation of only 82 parts per 1000. It is concluded from a comparison of the two expansion ratios that no significant adsorption of nitrogen on stainless steel occurs on decreasing the temperature from 303° K to 195° K in the pressure range 10^{-8} to 10^{-7} torr.

It should be noted that the expansion ratio for nitrogen at 303° K is significantly higher than the value of 1700 reported previously (ref. 1). Also the expansion ratios for argon and nitrogen at 303° K are significantly different. Both discrepancies can be explained on the basis of the gage pumping. In the previous work, the effect of gage pumping was minimized by an extrapolation procedure. In the present work, calculations were based on pressure 120 seconds after step 3 in the adsorption procedure. Using the results of Byvik and Bradford (ref. 3) for the pumping speed of nitrogen by a Varian nude gage, it can be shown that 90 percent of the observed decrease in the pressure of nitrogen in the adsorption vessel is due to gage pumping. The calculated extent of decrease is within the experimental error of the expansion ratio measurements. Therefore the larger expansion ratio for nitrogen in the present work can be attributed almost singly to gage pumping. Hence the results obtained in the previous and present work are reconciled on the basis of gage pumping. Similarly, at a given emission current, argon presumably has a lower pumping speed than nitrogen. The expansion ratios differ by a factor of 15 and thus would suggest that the gage pumping of these two gases differ by the same factor which is reasonable.

The results of this program have led to a detailed analysis of possible modifications in the experimental apparatus to enhance its capabilities. The suggested modifications are described in the appendix.

<center>CONCLUSION</center>

The following conclusion resulted from an experimental investigation of the adsorption of argon and nitrogen on stainless steel at 195° K and 303° K in the pressure range 10^{-8} to 10^{-7} torr:

1. Adsorption of nitrogen does not occur to a significant extent on stainless steel on decreasing the temperature from 303° K to 195° K in the pressure range 10^{-8} to 10^{-7} torr.

ACKNOWLEDGEMENTS

REFERENCES

1. Wightman, J. P.: The Adsorption of Selected Gases on Stainless Steel at Room Temperature in the Pressure Range 10^{-8} to 10^{-7} Torr. NASA-ASEE Summer Institute Report, Langley Research Center, 1965.

2. Maliakal, J. S.; and Herb, R. G.: Orbitron-New Principle in Ion Pumping. Research Development, June 1966, pp. 54-57.

3. Byvik, C. E.; and Bradford, J. M.: Pumping of Common Gases by Ionization Gauges Used in Space Simulation Facilities. Paper presented at AIAA/IES/ASTM Space Simulation Conference, September 1966.

APPENDIX

The following modifications to the present ultrahigh vacuum adsorption apparatus would significantly enhance its capabilities:

1. Varian nude gages (UHV-14) mounted on both the adsorption and storage vessels. The second gage on both vessels functions as a standby. This modification allows the necessary pumping speed data for a given gas to be obtained on the same gage type.

2. Mate to both vessels, coils compatible with various refrigerants including liquid nitrogen. This would extend temperature capability of system and allow measurements of adsorption thermodynamics.

3. Replace valve (V_1) with minimal gas load valve, perhaps Granville-Phillips type.

4. Replace valves (V_2) and (V_3) with either Nupro "BW" Series or Nupro "H" Series. Both are integrally welded bellows type valves.

5. Add a valve between valve (V_2) and storage vessel (SV) to permit measurement of contribution of (1-3) cycle during series of adsorption runs without having to evacuate storage vessel. The distances between the added valve and (SV) and (V_2) should be as small as possible.

TABLE I.- DESCRIPTION OF SYSTEM COMPONENTS

Designation	Item	Manuf.	Cat. No. (model)	Serial No.	NASA No.
NG_1, NG_2	Nude gage	Varian	UHV-14	--	--
--	Controller	Varian	971-0003	--	123946
TG_1, TG_2	Tub. gage	Veeco	RG75K	--	--
--	Controller	Veeco	RG21X	--	125592
$F_1 \rightarrow F_9$	Flange	Varian	954-5071	--	--
F_{10}, F_{11}	Flange	Ultek	48-156	--	--
F_{12}	Flange	Ultek	48-800	--	--
F_{13}, F_{14}	Flange	GE	22HF115	--	--
AV	Adsorption vessel	--	--	--	--
SV	Storage vessel	--	--	--	--
D	Doser	--	--	--	--
V_1, V_2	Valves	Nupro	SS4BG	--	--
V_3	Inlet valves	Varian	951-5100	--	--
V_4, V_5	Valve	Varian	951-5027	--	--
V_6	Valve	GE	22HV012	61043	--
V_7	Valve	GE	22HV012	50135	--
V_8	Valve	Veeco	--	--	--
T	Chamber	--	--	--	--
SP_1, SP_2	Sorption pump	GE	22HP111	--	--
OIP	Orb-ion pump	NRC	0206	0206K037	134711-1
--	Controller	NRC	254	K055	134712
TC	Thermocouple gage	Hastings	DV6M	--	--
--	Controller	Stokes	MB-3M	--	121362
Tr	Trap	--	--	--	--
Ta	Tank	--	--	--	--
P	Mech. pump	Welch	--	--	--
--	Recorder	Bristol	1P12G560-21-B14-T111	--	105910
--	Recorder	Sanborn	150-1000	--	112287
--	Recorder	Sanborn	150-1500	--	112286

TABLE II.- EXPANSION RATIOS FOR ARGON

Date	Run No.	T_{AV}, °K	P_B	P_{14}	P_{AV} (torr)	P_{AV}^{corr}	P_{SV}	R
8-5-66	1	303	4.6×10^{-10}	4.2×10^{-9}	3.15×10^{-8}	2.73×10^{-8}	4.0×10^{-5}	1450
	2	303	5.0	4.6	3.35	2.89	4.4	1500
	3	303	10.0	9.5	41.0	38.5	55.0	1450
8-8-66	1	303	3.3×10^{-10}	2.2×10^{-9}*	2.3×10^{-8}	2.1×10^{-8}	2.6×10^{-5}	1250
	2	303	3.8	3.3	3.65	3.32	4.5	1350
	3	303	4.0	3.5	5.2	4.85	6.4	1300
	4	303	14.0	13.5	7.4	6.05	8.4	1400
	5	303	22.0	21.0	23.5	21.4	31.0	1450
8-19-66	1	195	3.4×10^{-10}	9.5×10^{-10}*	2.75×10^{-8}	2.65×10^{-8}	3.3×10^{-5}	1250
	2	195	3.4	9.5	5.2	5.1	6.3	1250
	3	195	3.6	9.8	6.7	6.6	7.8	1200
	4	195	3.8	10.0	20.5	20.4	33.0	1600
	5	195	4.1	11.0	41.5	41.4	63.0	1500

*Directly measured values.

TABLE III.- EXPANSION RATIOS FOR NITROGEN AT 303° K

Date	Run No.	P_B	P_{14}	P_{AV} (torr)	P_{AV}^{corr}	P_{SV}	R
8-2-66	1	3.3×10^{-10}	3.0×10^{-9}	4.7×10^{-9}	1.7×10^{-9}	2.7×10^{-5}	16000
	2	4.0	3.5	5.8	2.3	3.7	16000
	3	4.0	3.5	6.6	3.1	5.7	18500
	4	4.0	3.5	7.8	4.3	7.1	16500
	5	4.4	3.8	15.0	11.0	23.0	21000
	6	5.0	4.4	26.0	22.0	45.0	20000
	7	5.8	5.2	30.0	25.0	59.0	23500
	8	7.1	6.5	36.0	30.0	76.0	25000
8-3-66	1	4.7×10^{-10}	4.1×10^{-9}	3.8×10^{-8}	3.4×10^{-8}	7.4×10^{-4}	22000
	2	5.0	4.4	2.6	2.2	6.2	28000
	3	5.2	4.6	2.2	1.7	4.6	27000
	4	5.8	5.2	1.6	1.1	3.2	29000
8-4-66	1	2.6×10^{-10}	2.1×10^{-9}	3.2×10^{-9}	1.1×10^{-9}	1.5×10^{-5}	13500
	2	3.1	2.5	4.3	1.8	3.7	20500
	3	3.2	2.7	4.8	2.1	5.7	27000
	4	3.6	3.1	12.0	9.0	20.0	22000
	5	4.4	3.8	18.0	14.0	40.0	28500
	6	4.6	4.0	28.0	24.0	58.0	24000
	7	5.0	4.5	32.0	28.0	79.0	28000

TABLE IV.- EXPANSION RATIOS FOR NITROGEN AT 195° K

Date	Run No.	P_B	P_{14}	P_{AV} (torr)	P_{AV}^{corr}	P_{SV}	R
8-11-66	1	6.3×10^{-10}	1.2×10^{-9}*	1.20×10^{-9}	0.0	2.7×10^{-5}	---
	3	6.6	1.3	11.5	10.0×10^{-9}	29.0	29000
	4	7.2	1.9	28.0	26.0	59.0	22500
8-11-66(A)	1	6.2×10^{-10}	1.2×10^{-9}*	2.2×10^{-9}	1.0×10^{-9}	2.7×10^{-5}	27000
	2	6.4	1.5	3.5	2.0	5.4	27000
	3	6.2	1.2	4.8	3.6	8.0	22000
	4	6.6	1.6	13.0	11.0	30.0	27000
	5	6.9	1.7	25.0	23.0	57.0	25000
8-12-66	1	6.1×10^{-10}	1.3×10^{-9}	2.5×10^{-9}	1.2×10^{-9}	2.7×10^{-5}	21500
	2	6.1	1.3	3.5	2.2	5.3	24000
	3	6.3	1.4	4.7	3.3	7.6	23000
	4	6.6	1.6*	11.0	9.4	2.4	25500
	5	7.0	1.8	26.0	24.0	6.0	25000
	6	7.5	2.2	38.0	36.0	7.8	21500

*Directly measured values.
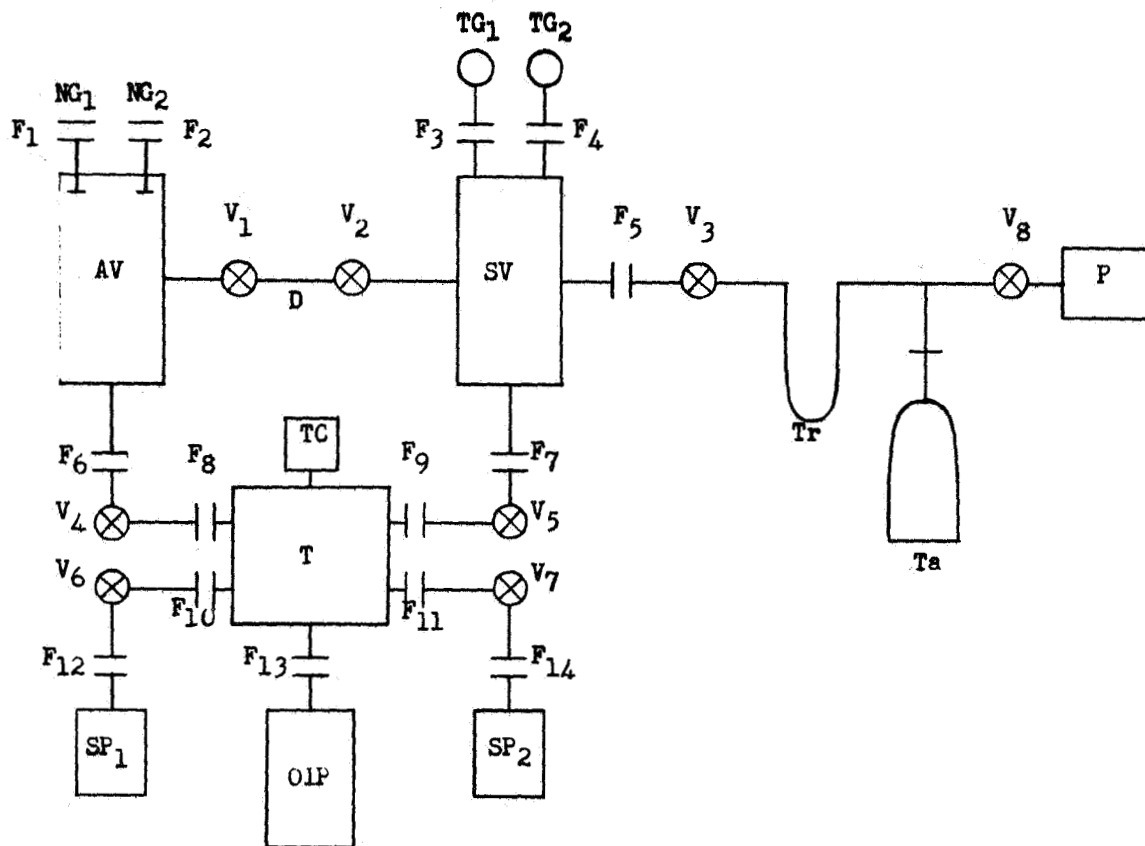
Figure 1.- Schematic of ultrahigh vacuum apparatus. (See table 1 for symbol identification)
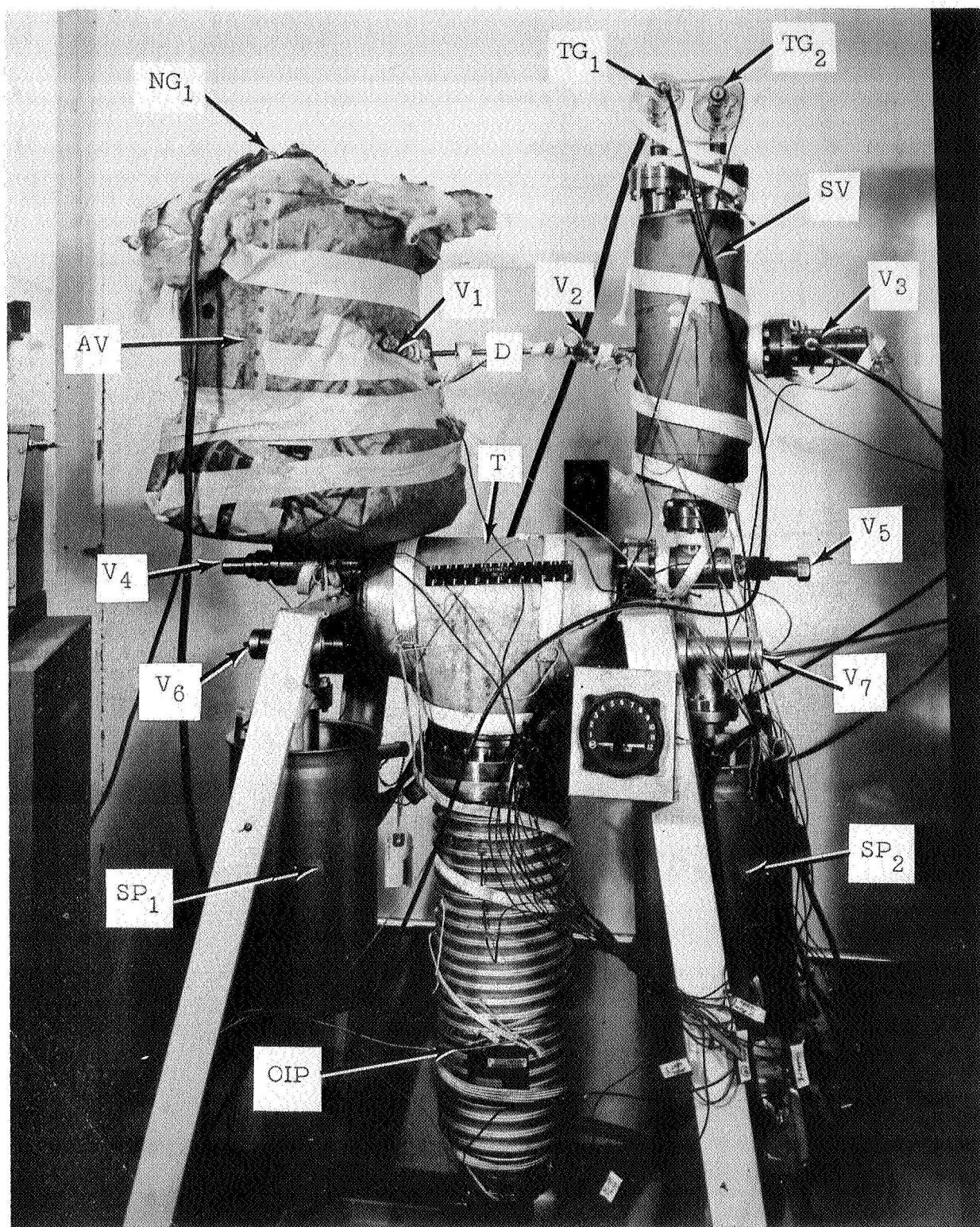
Figure 2. - Ultrahigh vacuum adsorption apparatus.
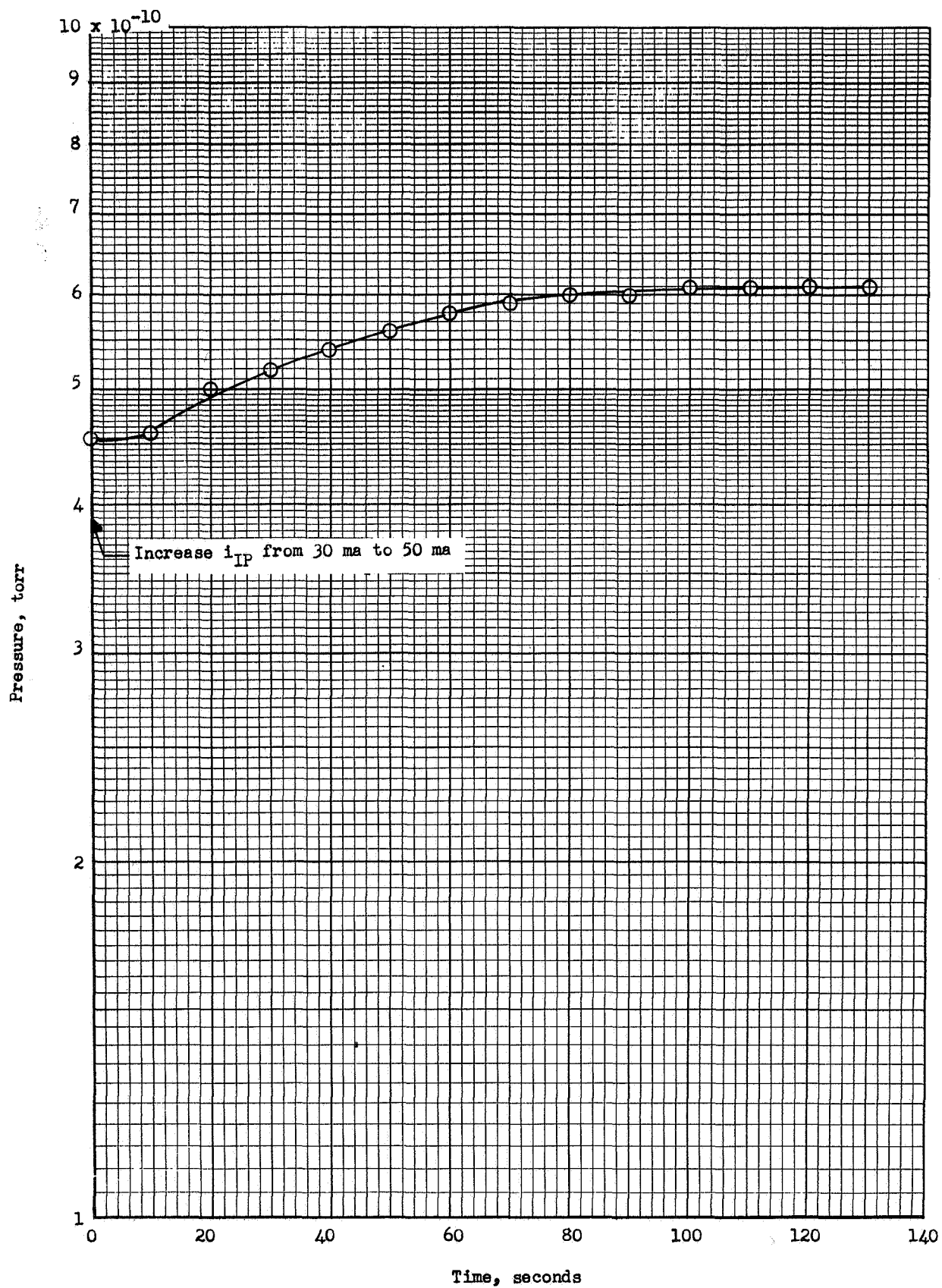
Figure 3.- Effect of change in ion pump emission current (i_{IP}) on pressure in adsorption vessel [Bias: 8, Emission: 8, Varian Gage (NG₁): $i^- = 4$ ma]
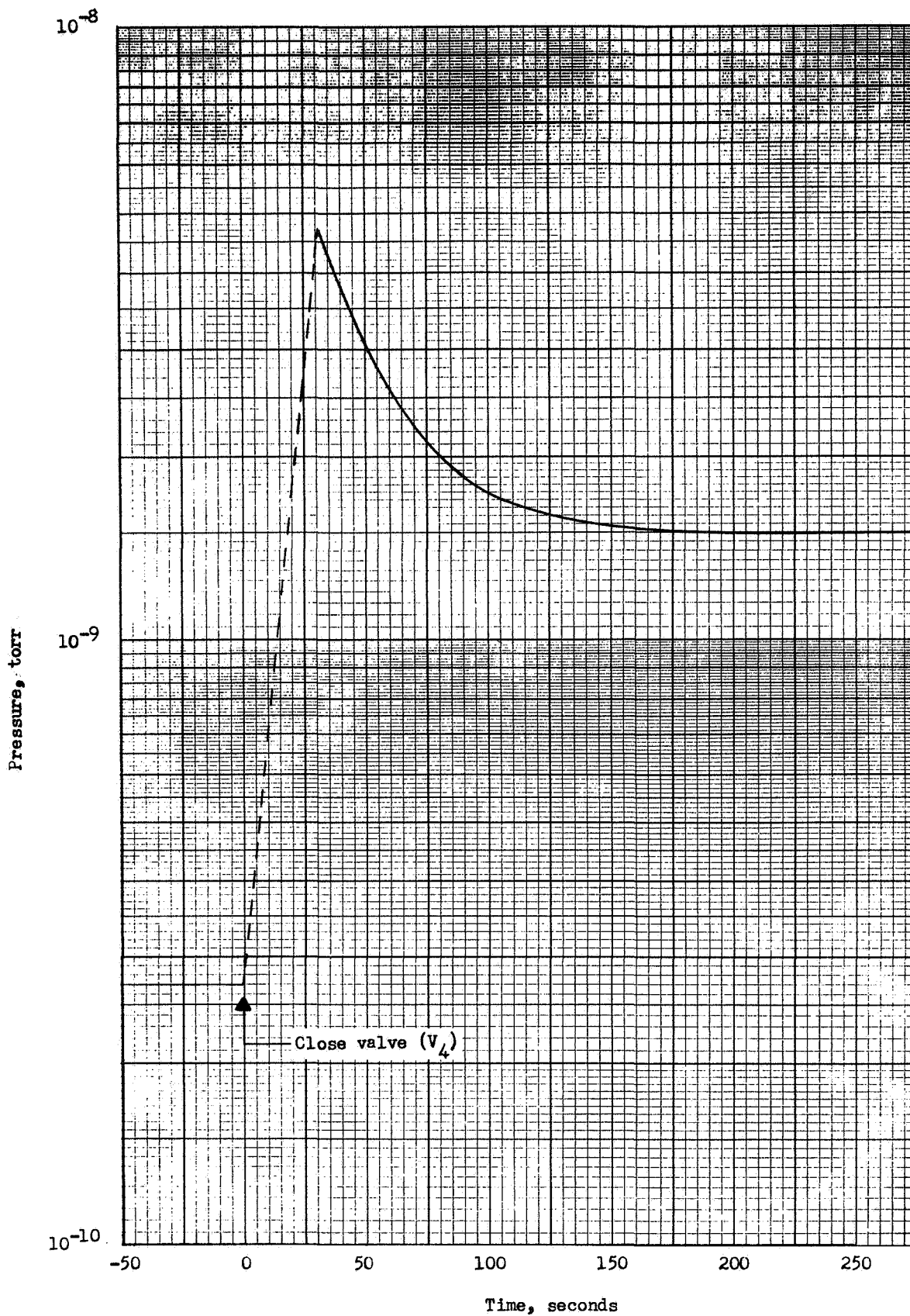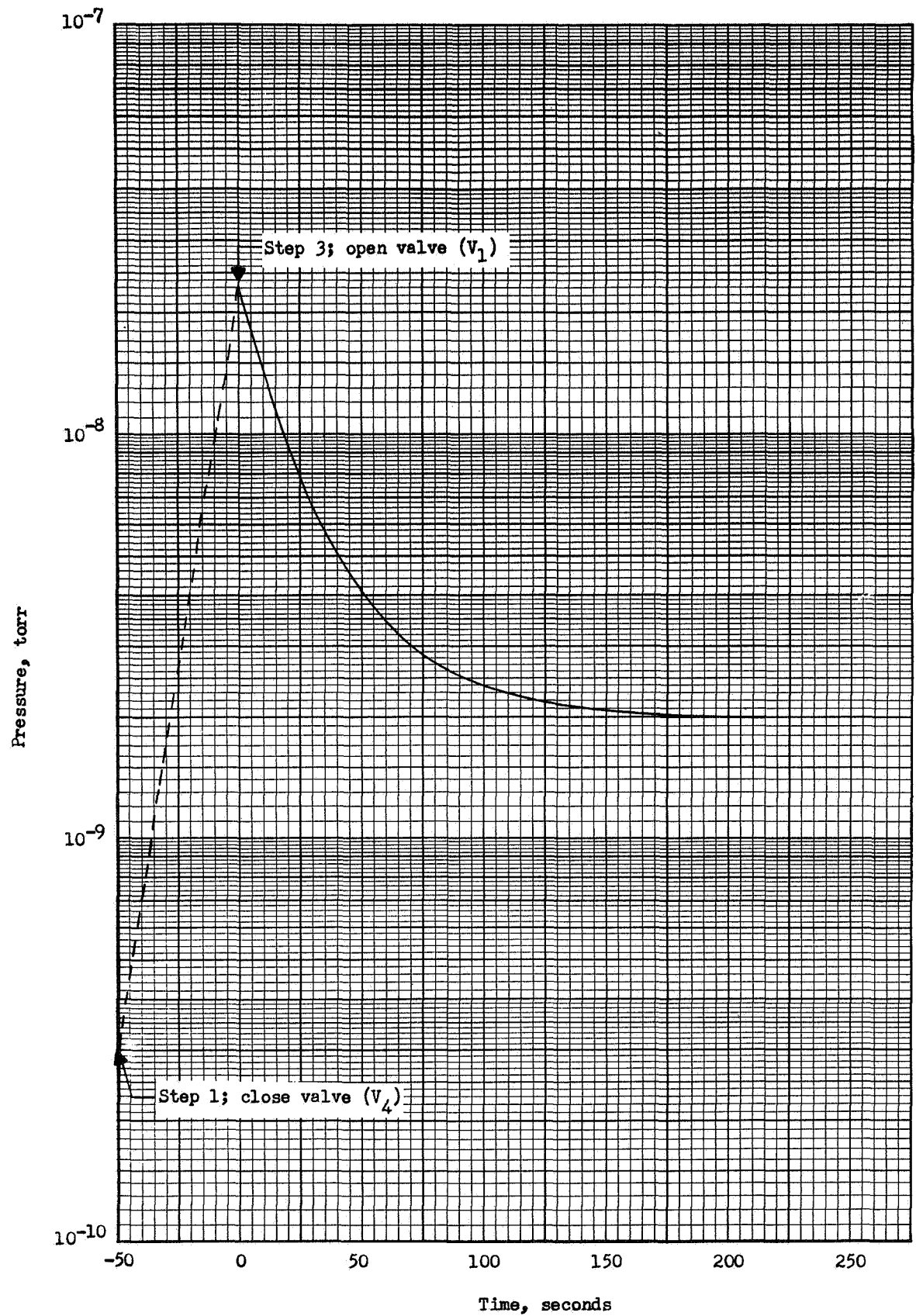
Figure 4.- Typical pressure-time trace on closing valve (V_4). Data of 8-5-66 (run no. 1)
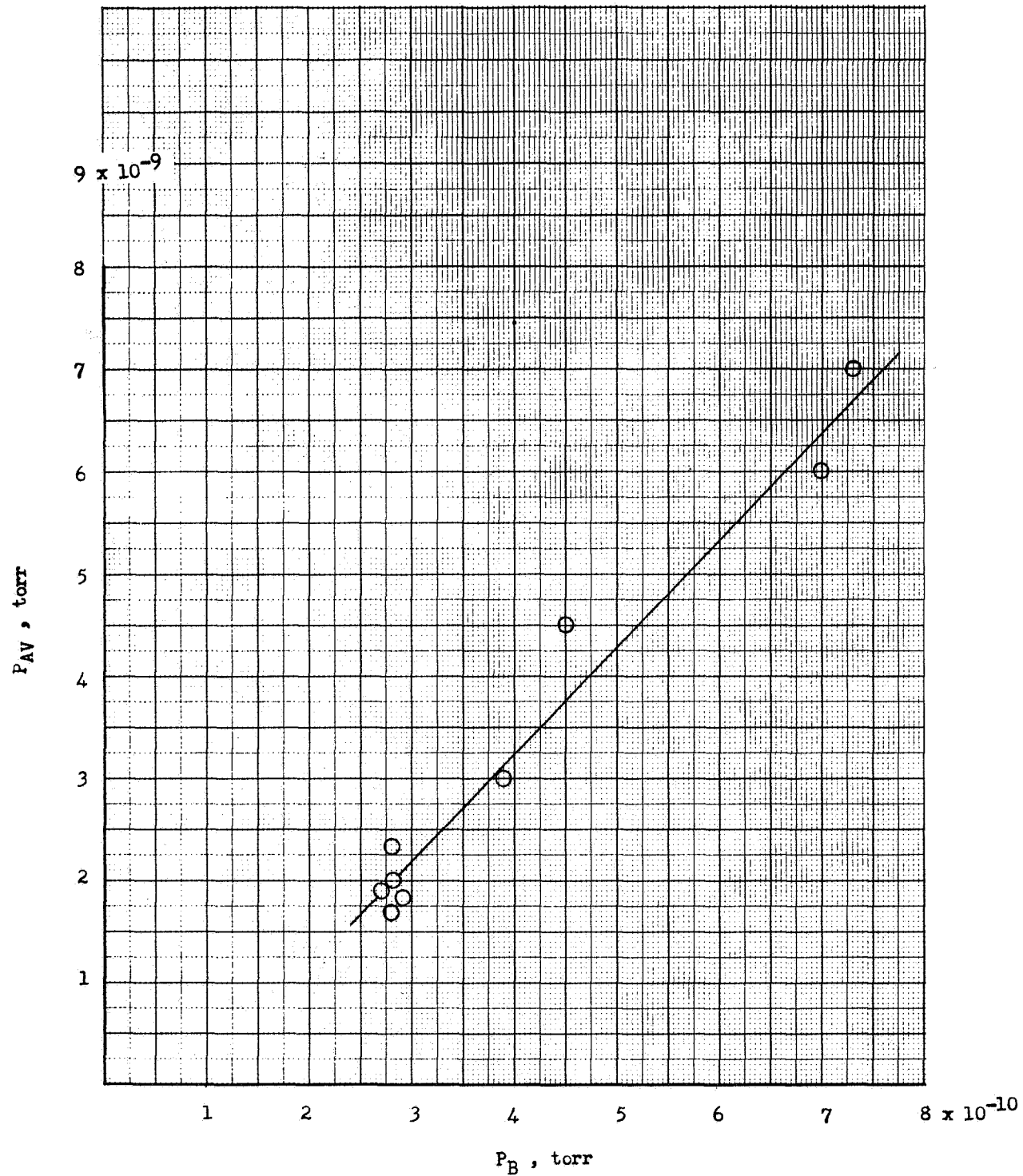
Figure 5.- Pressure in adsorption vessel (P_{AV}) at 303°K and 120 seconds after closing valve (V_4) as a function of base pressure (P_B) in adsorption vessel prior to closing valve (V_4).

Figure 6.- Pressure in adsorption vessel (P_{AV}) at 195°K and 120 seconds after closing valve (V_4) as a function of base pressure (P_B) in adsorption vessel prior to closing valve (V_4).

Figure 7.— Typical pressure-time trace for (1-4) cycle. Data of 8-5-66 (run no. 1)
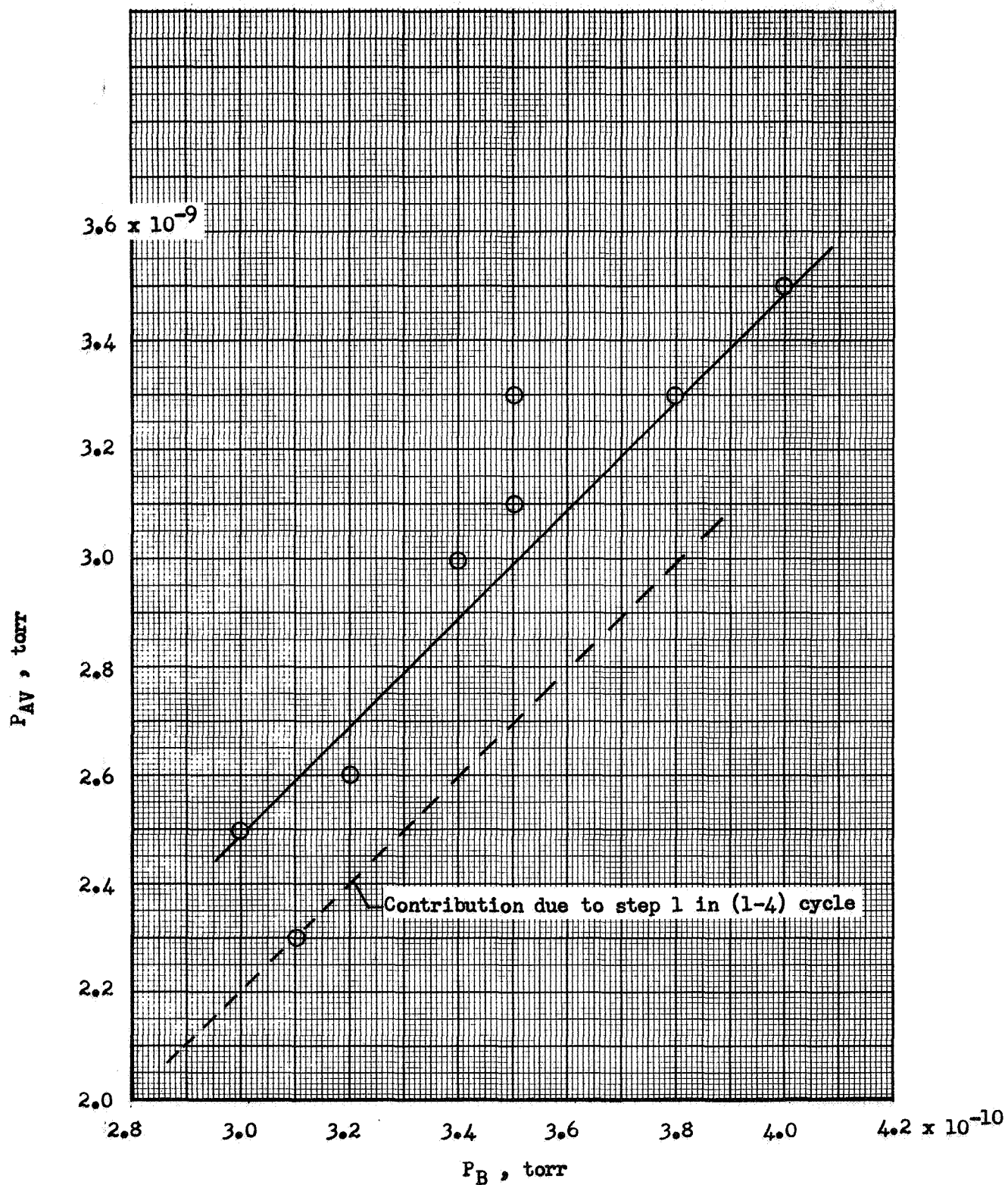
Figure 8.- Pressure in adsorption vessel (P_{AV}) at $303^{\circ}K$ and 120 seconds after opening valve (V_1) - step 3 in (1-4) cycle as a function of base pressure (P_B) in adsorption vessel prior to initiation of (1-4) cycle.
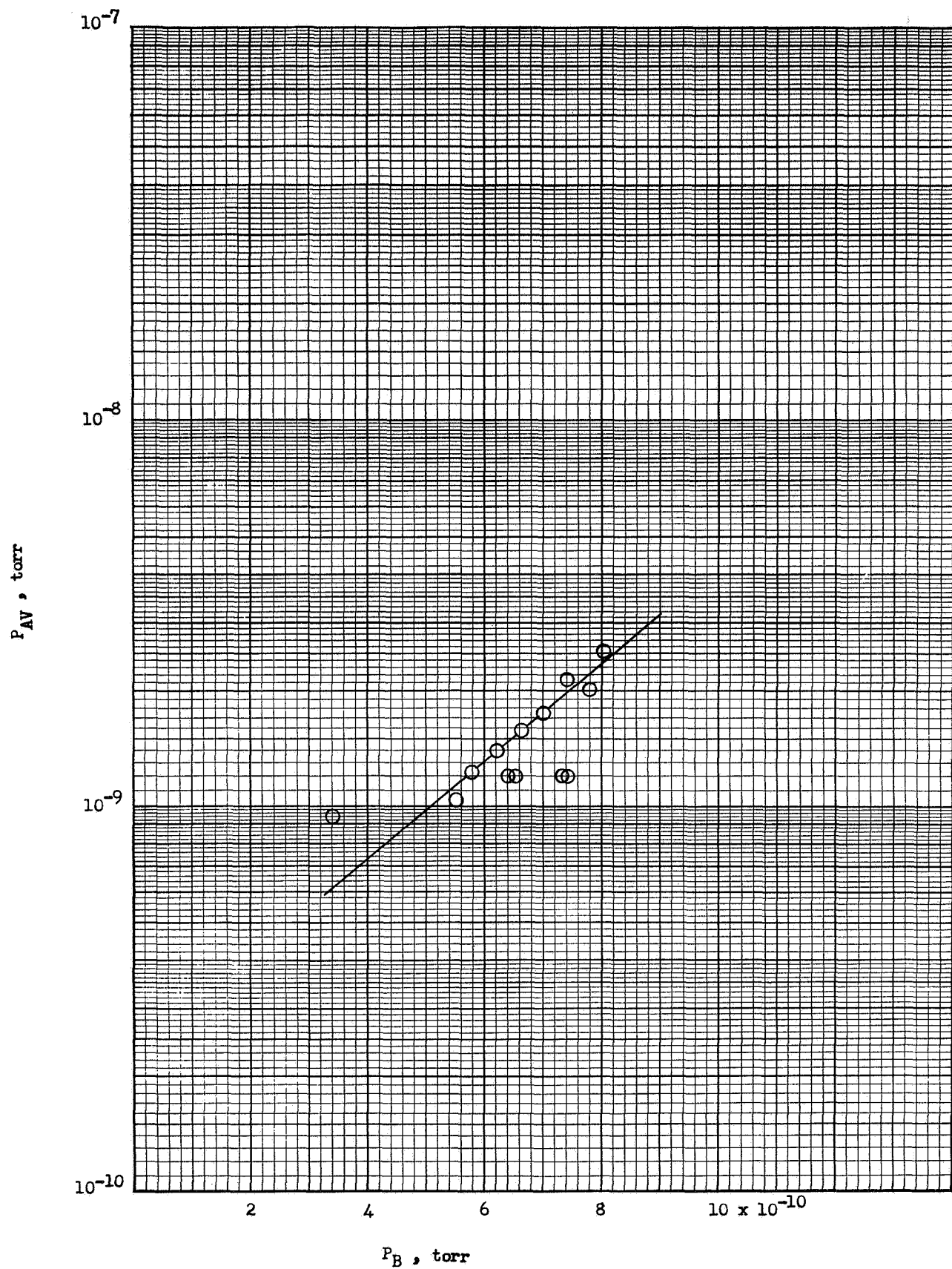
Figure 9.- Pressure in adsorption vessel (P_{AV}) at 195° K and 120 seconds after opening valve (V_1) - step 3 in (1-4) cycle - as a function of base pressure (P_B) in adsorption vessel prior to initiation of (1-4) cycle.
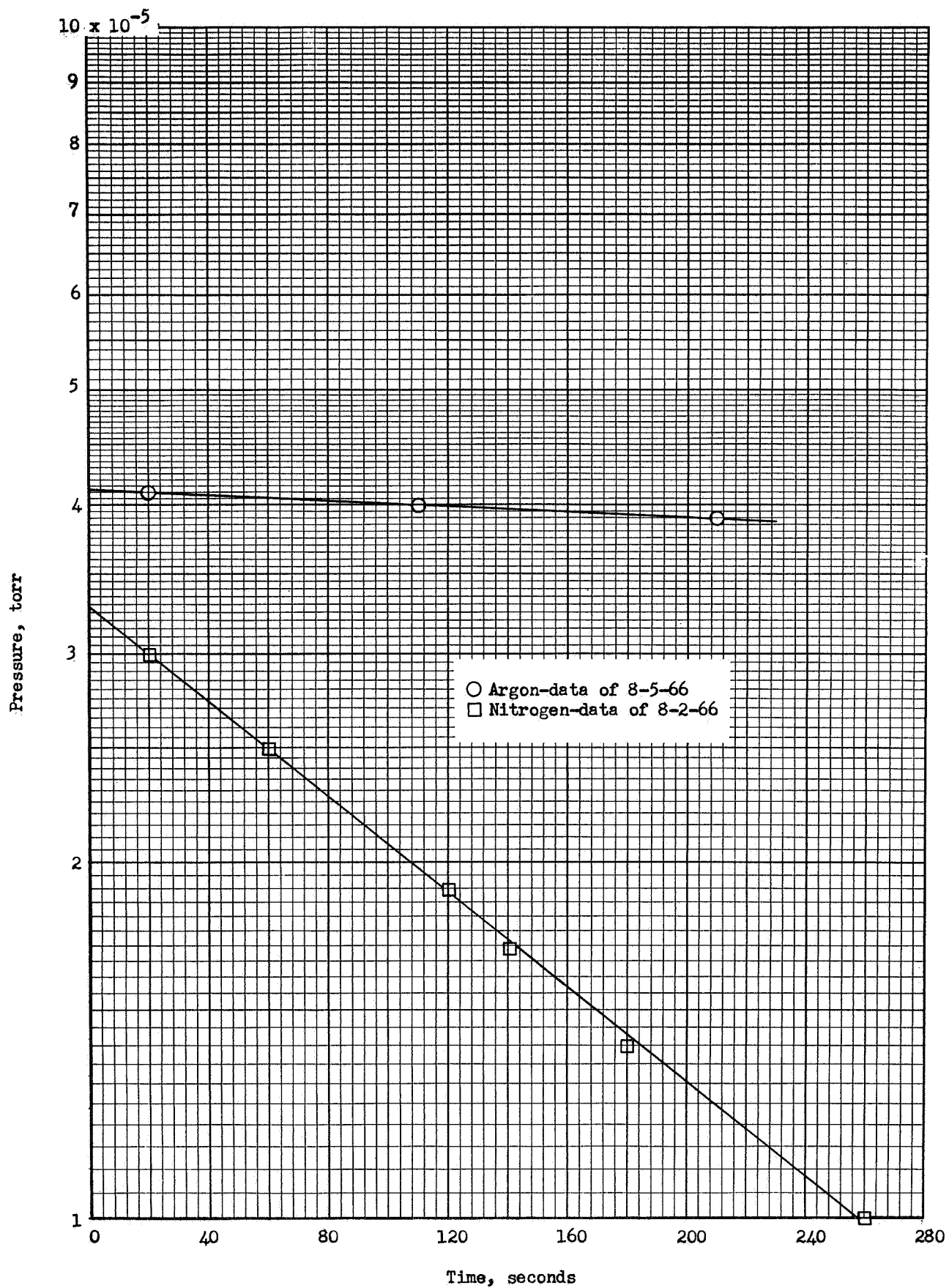
Figure 10.- Pressure in storage vessel with Veeco gage (TG_1) at 1 ma emission as a function of time.
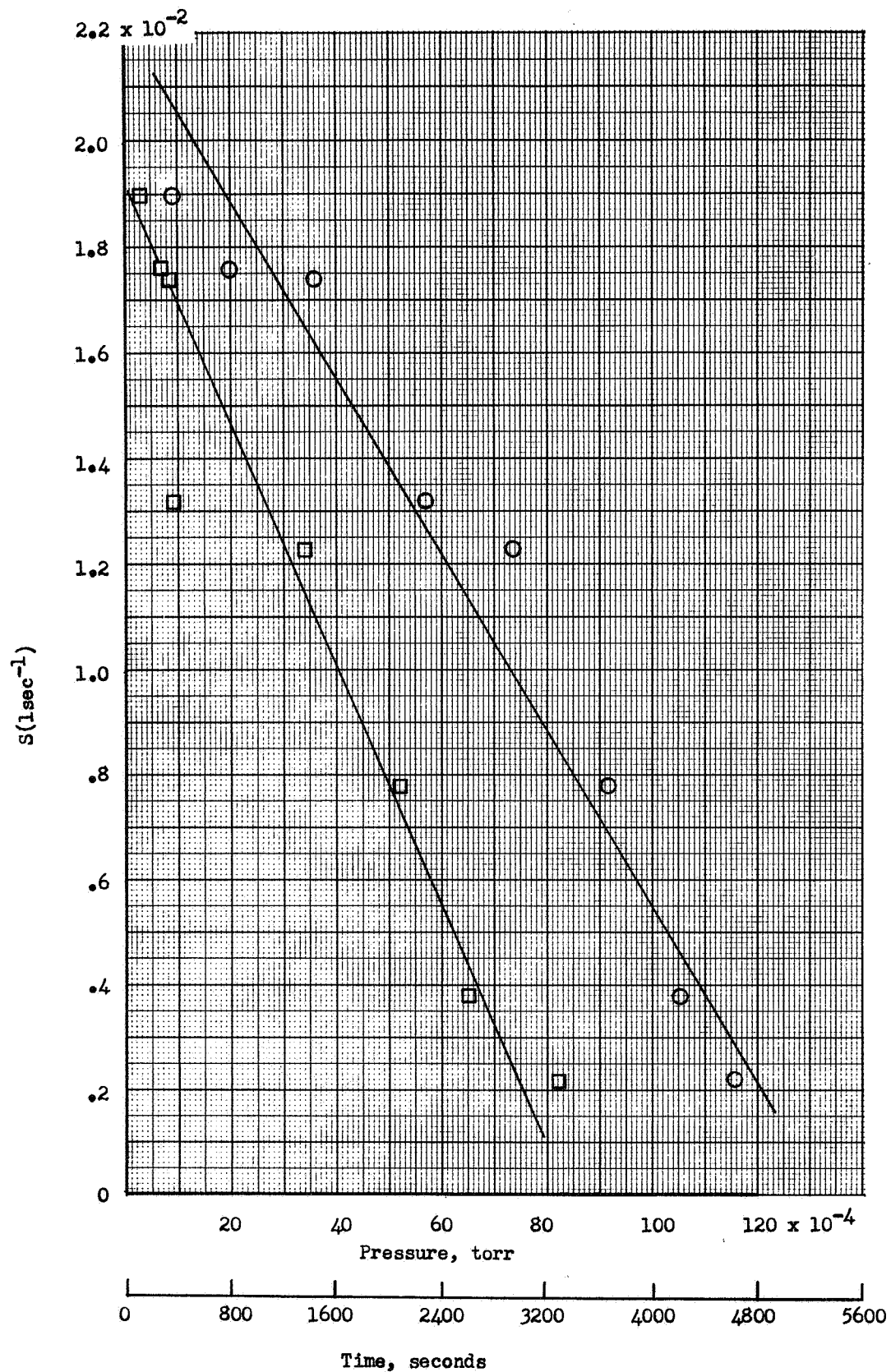
Figure 11.- Pumping speed of nitrogen by gage (TG_1) at 1 ma emission as a function of nitrogen pressure (□) and time after closing valve (V_5) (O). Data of 8-2-66
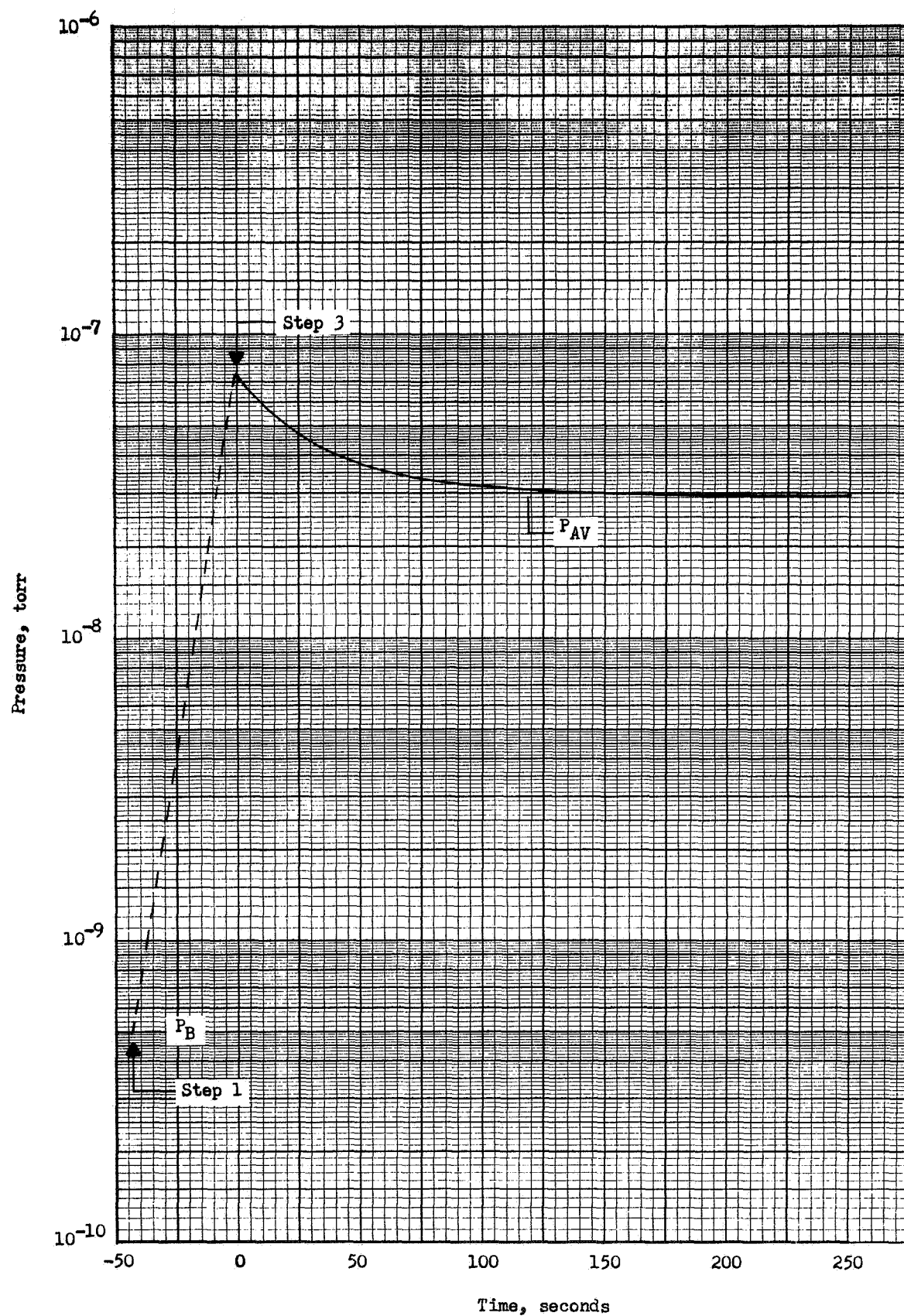
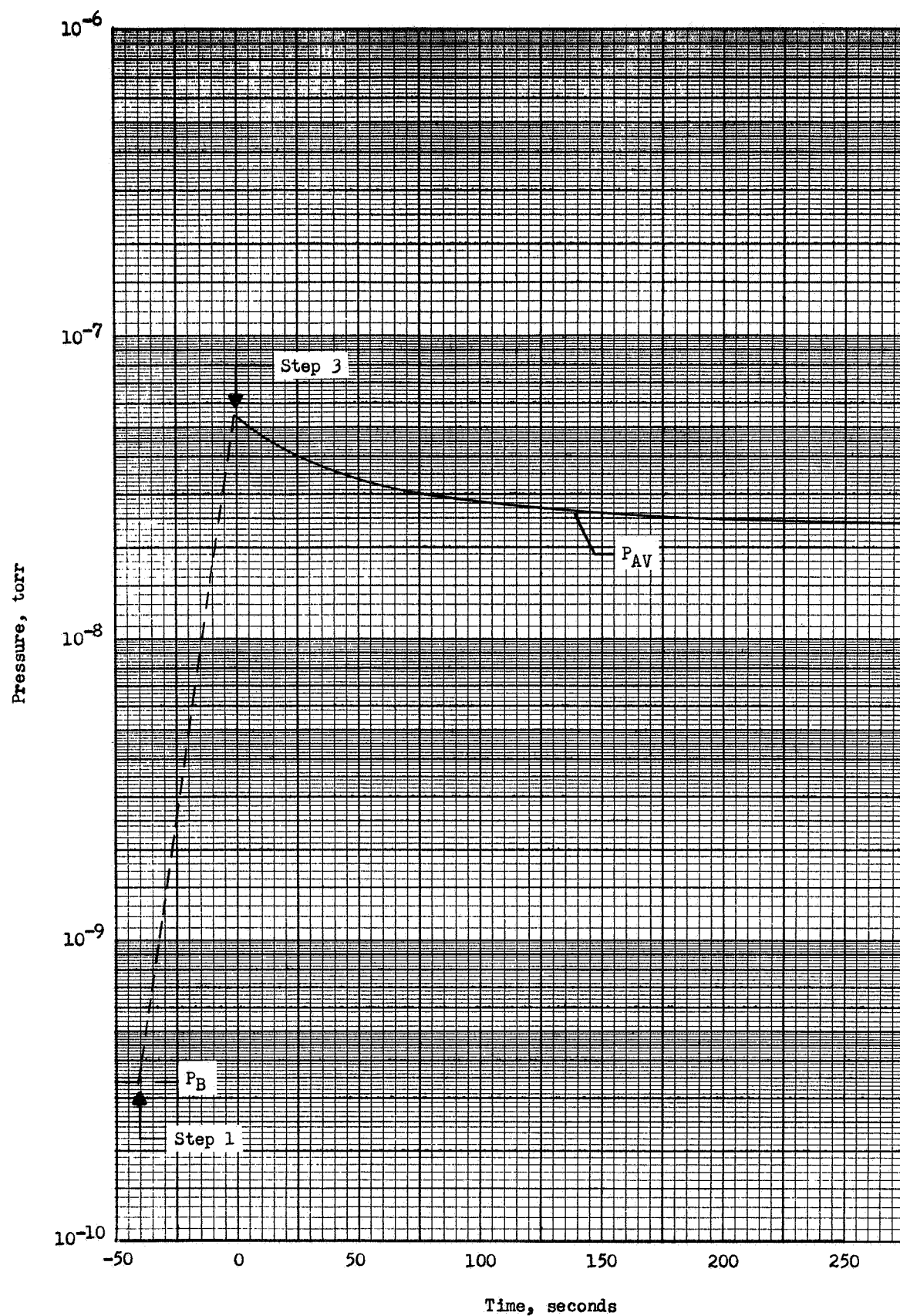Figure 12 (a).- Typical pressure-time trace for the expansion ratio of argon at 303° K. Data of 8-5-66 (run no. 2)

Figure 12 (b).- Typical pressure-time trace for the expansion ratio of argon at 195° K.
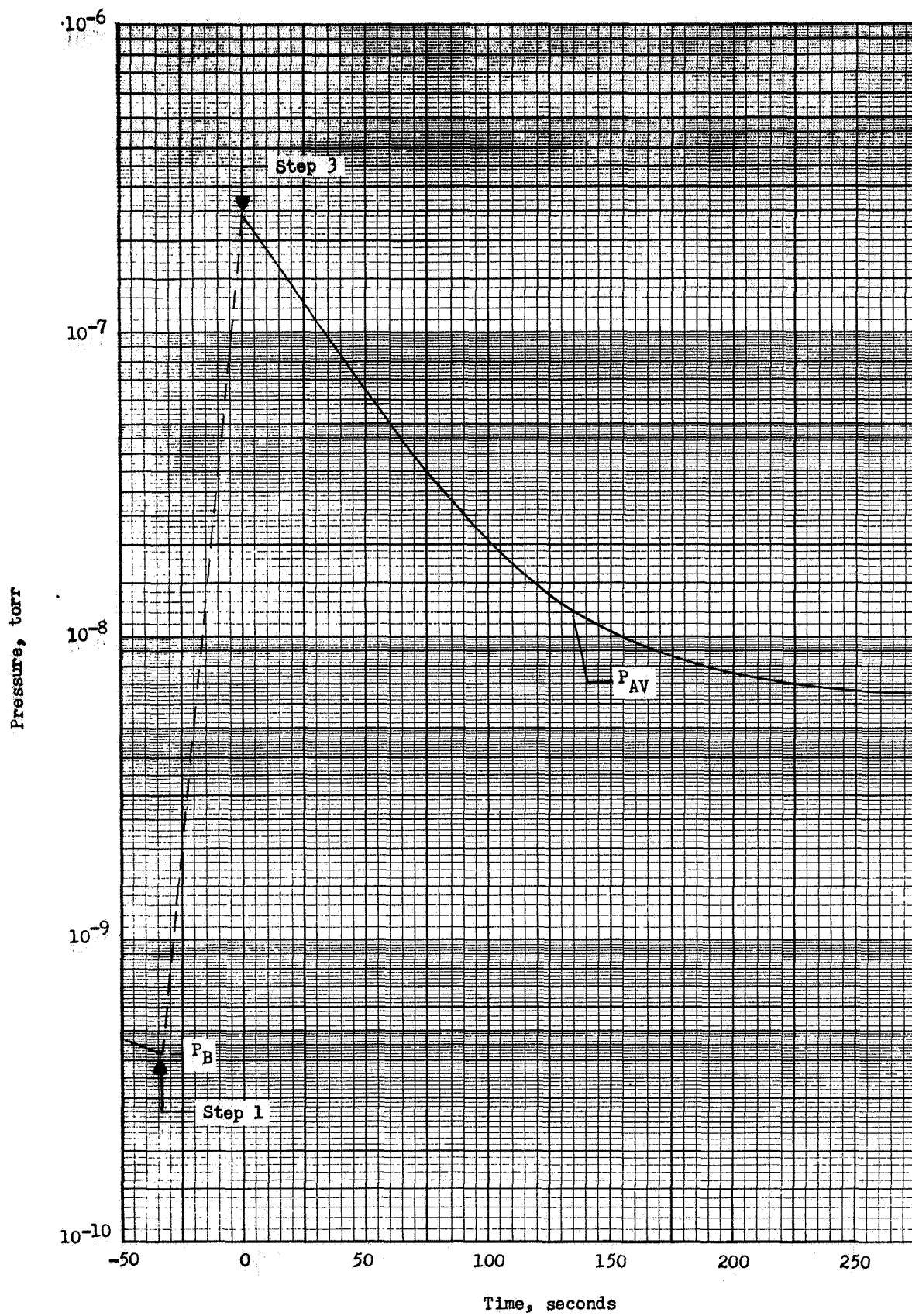Data of 8-19-66 (run no. 1)

Figure 13 (a).- Typical pressure-time trace for the expansion ratio of nitrogen at 303° K. Data of 8-2-66 (run no. 5)
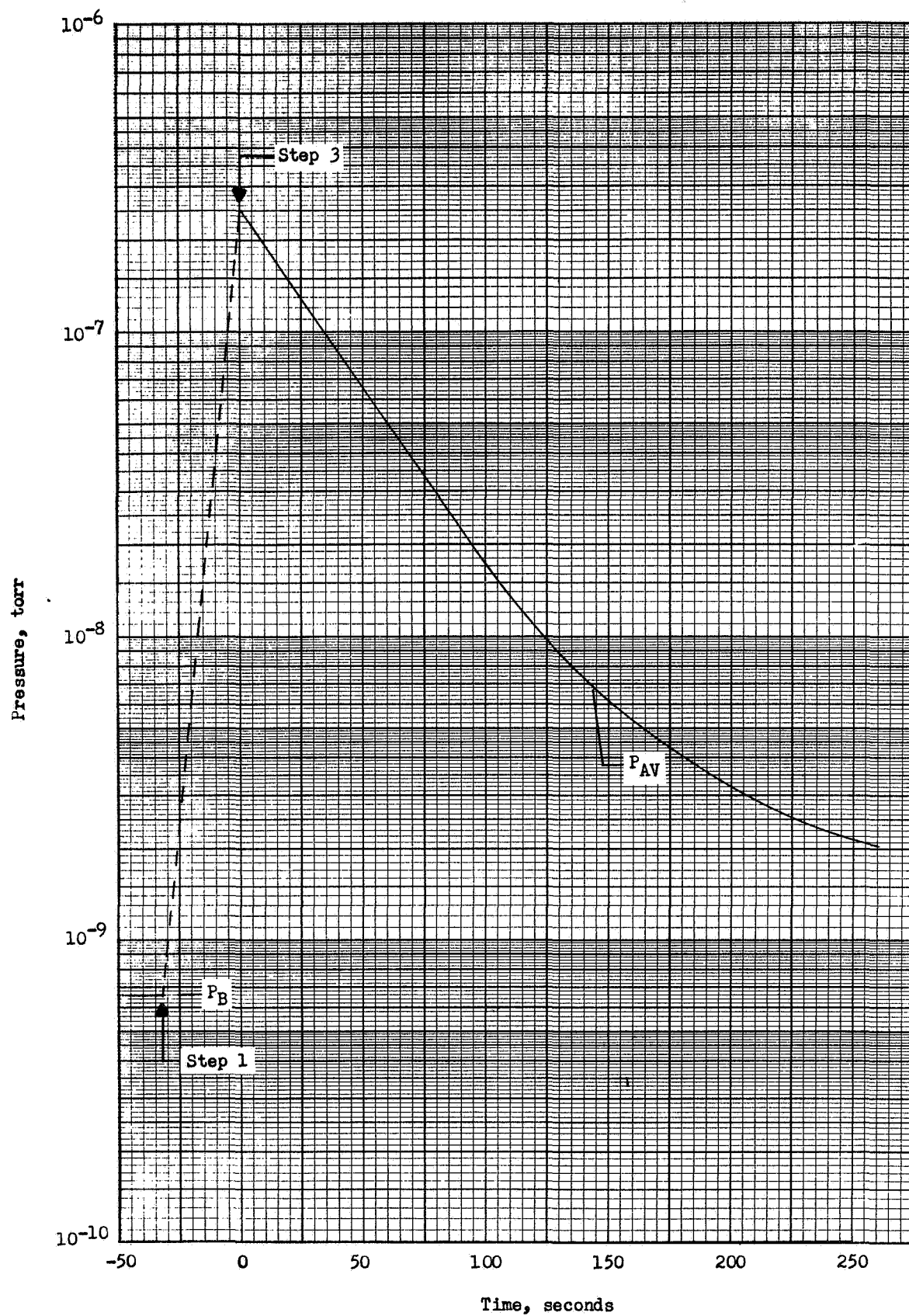
Figure 13 (b).- Typical pressure-time trace for the expansion ratio of nitrogen at 195° K.
Data of 8-12-66 (run no. 4)